ALS Laboratory Group
ANALYTICAL CHEMISTRY & TESTING SERVICES

Nonhalogenated Organic Analysis

by GC/FID

(EPA Method 8015)

Date: 04/01/2009

APPROVED BY: _____ DATE: 3-26-09
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Analysis of Nonhalogenated Organics by GC/FID

1) SCOPE AND APPLICABILITY

1.1 This SOP is used to determine the concentration of various volatile and semivolatile organic compounds by gas chromatography. The following compounds can be determined by this method.

Table 1.1

Compound Name	CAS No.			
1-Butanol	71-36-3			
t-Butanol	75-65-0			
Ethanol	64-17-5			
Ethylene glycol	107-21-1			
Isobutanol	78-83-			
Isopropanol	67-63-0			
Methanol	67-56-1			
1-Propanol	71-23-8			
Propylene Glycol	57-55-6			
Gasoline range organics				
Diesel range organics				
Organic range organics				

1.2 This method may also be applicable to the analysis of petroleum hydrocarbons, including gasoline range organics (GRO), diesel range organics (DRO), and oil range organics (ORO). GROs correspond to the range of alkanes from C6 to C10, covering a boiling range of approximately 60 C – 170 C. DROs correspond to the alkanes from C10 to C28, covering the boiling range from 170 C – 430 C. OROs correspond to the alkanes over C28.

1.3 This method may be used for any analyte that could be chromatographically separated and that gives an adequate response by FID.

1.4 This method is restricted for use by, or under the supervision of, analysts experienced in the use of gas chromatographs and skilled in the interpretation of gas chromatograms. In addition, if this method is used for the analysis of petroleum hydrocarbons, it is limited to analysts experienced in the interpretation of hydrocarbon data. Each analyst must demonstrate the ability to generate acceptable results with this method.

1.5 The method may also be used as a screening tool (for both volatile and semivolatile organics) to obtain semiquantitative data for the prevention of sample overload during quantitative analysis on a GC/MS system.

2) SUMMARY OF METHOD

2.1 Method 8015 provides gas chromatographic conditions for the detection of certain volatile and semivolatile organic compounds.

2.1.1 Samples may be introduced to the GC following solvent extraction (methods 3510, 3540, etc.).
2.1.2 Ground waters and 1:1 extracted soils may be directly injected into the GC.
2.1.3 DROs and OROs must be prepared using an appropriate solvent extraction method.

2.2 An appropriate column and temperature program is used in the gas chromatograph to separate the organic compounds. Analyte detection is achieved with a flame ionization detector (FID).

2.3 The method allows the use of capillary columns for the analysis and confirmation of the individual analytes. Columns and conditions listed have been demonstrated to provide separation of those target analytes. Analysts may change these conditions as long as they demonstrate adequate performance.

3) DEFINITIONS

3.1 GC/FID: Gas Chromatograph / Flame Ionization Detector

3.2 Organic Free Water: Deionized (DI) reagent water meeting purity characteristic of ASTM Type II laboratory distilled water (daily conductivity <1.0 umhos/cm). For additional purification before use, the DI water is passed through an activated carbon filter.

3.3 Laboratory Control Sample (LCS): A known matrix spiked with compound(s) representative of the target analytes and used to evaluate/document laboratory method performance.

3.4 Laboratory Control Sample Duplicate (LCSD): A known, clean matrix spiked with compound(s) representative of the target analytes and used to evaluate/document laboratory performance. This sample is duplicate of the LCS.

3.5 Matrix: The component or substrate (e.g., surface water, groundwater, soil) containing the analyte(s) of interest.

3.6 Matrix Spike (MS): An aliquot of sample spiked with a known concentration of target analyte(s) prior to sample extraction and processing. The MS is used to evaluate bias of a method in a given sample matrix.

3.7 Matrix Spike Duplicate (MSD): A duplicate sample spiked with identical concentrations of target analyte(s) prior to sample extraction and processing. The MS/MSD pair is used to assess the precision and bias of a method in a given sample matrix.

3.8 Method Blank: An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank is carried through the complete sample preparation and analytical procedure. The method blank is used to document the presence/absence of contamination as a result of the analytical process.

3.9 Standard Curve: A plot of known analyte concentrations (standards) versus the instrument response.

3.10 CAR: Corrective Action Report (refer to SOP HS-QS-003, current revision).

4) HEALTH AND SAFETY WARNINGS

4.1 Lab Safety: Due to various hazards in the laboratory, safety glasses and laboratory coats or aprons should be worn at all times while in the laboratory. In addition, gloves and a face shield should be worn when dealing with toxic, caustic, and/or flammable chemicals.

4.2 Chemical Hygiene: The toxicity or carcinogenicity of each reagent used has not been precisely defined; however, each chemical used should be treated as a potential health hazard. Exposure to laboratory reagents should be reduced to the lowest possible level. The laboratory maintains a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of data handling sheets (MSDS) is available to all personnel involved in these analyses.

4.3 Waste Disposal: Extracts generated from this procedure are maintained in a segregated waste stream. It is the laboratory's responsibility to comply with all federal, state, and local regulations governing waste management by minimizing and controlling all releases from fume hoods and bench operations. Compliance with all sewage discharge permits and regulations is required.

4.4 Pollution Prevention: The materials used in this method pose little threat to the environment when recycled and managed properly. The quantities of chemicals purchased should be based on the expected usage during its shelf life. Standards and reagent should be prepared in volumes consistent with laboratory use to minimize the volume of expired standards or reagents to be

disposed.

5) CAUTIONS

5.1 Routine preventative maintenance must be performed as scheduled and documented to assure optimum instrument performance. Refer to HS-EQ-04 for additional information.

6) INTERFERENCES

6.1 When analyzing for volatile organics, samples can be contaminated by diffusion container septum during shipment and storage. A trip blank prepared from organic-free reagent water and carried through sampling and subsequent storage and handling must serve as a check on such contamination.

6.2 Contamination by carryover can occur whenever high-concentration and low-concentration samples are analyzed in sequence. To reduce the potential for carryover, the sample syringe or purging device must be rinsed out between samples with an appropriate solvent. Whenever an unusually concentrated sample is encountered, it should be followed by injection of a solvent blank to check for cross contamination.

6.21 All glassware must be scrupulously cleaned. Clean all glassware as soon as possible after use by rinsing with the last solvent used. Detergent washing with hot water, rinsing with tap water, and then organic-free reagent water, should follow this. Drain the glassware and dry in an oven at 130°C for several hours or rinse with methanol and drain. Store dry glassware in a clean environment.

6.2 The flame ionization detector (FID) is a non-selective detector. There is a potential for many non-target compounds present in samples to interfere with this analysis.

7) PERSONNEL QUALIFICATIONS

7.1 General Responsibilities - This method is restricted to use by or under the supervision of analysts experienced in the method.

7.2 Analyst - It is the responsibility of the analyst(s) to:

7.2.1 Produce contractually compliant data that meets all quality

requirements using this procedure and the Data Reduction, Review and Validation SOP.

7.2.2 Complete the required demonstration of proficiency prior to performing this procedure independently.

7.2.3 Create and populate a data entry batch in LIMS for review by the Supervisor (or designee).

7.3 Section Supervisor - It is the responsibility of the section supervisor to:

7.3.1 Ensure that all analysts have the technical ability and have received adequate training required to perform this procedure.

7.3.2 Ensure analysts have completed the required demonstration of proficiency prior to performing this procedure without supervision.

7.3.3 Produce contractually compliant data that meets all quality requirements using this procedure and the Data Reduction, Review and Validation SOP.

7.3 Project Manager - It is the responsibility of the Project Manager to ensure that all contractual requirements for a client requiring this procedure are understood prior to initiating this procedure for a given set of samples.

8) SAMPLE COLLECTION, HANDLING AND PRESERVATION

8.1 All samples must be iced or refrigerated at 4°C (+/- 2 °C) from the time of collection until extraction. Refer to Table 8.1 for sample containers, sample preservation and sample holding time information.

TABLE 8.1 – Volatile and semivolatiles compounds by GC/FID

Sample Matrix	Container	Preservative	Holding Time
Concentrated Waste Samples	125-ml widemouth glass with Teflon lined lid.	None	Samples extracted within 14 days and extracts analyzed within 40 days following extraction.
Aqueous Samples	Direct injection: 3 x 40ml vials Extractable: 1L glass amber, Teflon lined lid.	Cool to 4°C	Direct injection:14days DRO/ORO:Samples extracted within 7 days and extracts analyzed within 40 days following extraction.

Solid Samples (e.g. soils, sediments, sludges, ash)	250-ml widemouth glass container with Teflon-lined lid	Cool to 4°C	Direct injection following 1:1 extraction:14days DRO/ORO:Samples extracted within 14 days and extracts analyzed within 40 days following extraction.

9.0 APPARATUS AND MATERIALS

9.1 Gas chromatograph

91.1 Gas Chromatograph - Analytical system complete with gas chromatograph suitable for solvent injections and all required accessories, including detectors, column supplies, recorder, gases, and syringes. A data system for measuring peak heights and/or peak areas is recommended.

9.1.2 Recommended GC Columns

9.1.2.1 Column 1 - 30 m x 0.53 mm ID fused silica capillary column bonded with polyethylene glycol (DB-Wax or equivalent), 1-µm film thickness.

9.1.2.2 Column 2 - 30 m x 0.53 mm ID fused silica capillary column chemically bonded with 5% diphenyl / 95% dimethyl polysiloxane (DB-5, SPB-5, RTx, or equivalent), 1.5-µm film thickness.

9.1.2.3 Column 3 –105m x 0.53 mm ID fused silica capillary column chemically bonded with proprietary dipheny / dimethyl polysiloxane phase (Rtx-502.2), 3.0-µm film thickness.

9.1.2.2.1 Wide-bore columns should be installed in 1/4-inch injectors, with deactivated liners designed specifically for use with these columns.

9.1.3 Detector - Flame ionization (FID)

9.2 Sample introduction and preparation apparatus

9.2.1 Refer to the 5000 series sample preparation methods for the appropriate apparatus.

9.2.2 Samples may also be introduced into the GC via injection of solvent extracts or direct injection of aqueous samples.

9.3 Syringes

9.3.2 Microsyringes - 10- µL, 25-µL, 100-µL, 500-µL, and 1000-µL.

9.4 Volumetric flasks, Class A - Appropriate sizes with ground glass stoppers.

10) Reagents And Standards

10.1 Reagent grade (or higher) chemicals shall be used whenever possible. Other grades may be used, provided it is ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

10.2 Organic-free reagent water - All references to water in this method refer to organic-free reagent water, as defined in Chapter One.

10.3 Methanol (CH_3OH), Pesticide quality or equivalent. Store away from other solvents.

10.4 Stock standards - Stock solutions may be prepared from pure standard materials or purchased as certified solutions. When methanol is a target analyte or when using azeotropic distillation for sample preparation, standards should not be prepared in methanol. Standards must be replaced after 12 months or sooner, if comparison with check standards indicates a problem.

10.5 Secondary dilution standards - Using stock standard solutions, prepare secondary dilution standards, as needed. The secondary dilution standards should be prepared at concentrations suitable for analytical determinations. Secondary dilution standards should be stored with minimal headspace and should be checked frequently for signs of degradation or evaporation.

10.7 Calibration standards - Calibration standards at a minimum of five different concentrations are prepared in water (direct injection) or in methylene chloride (solvent injection) from the secondary dilution of the stock standards. One of the standards should be at or below the concentration equivalent to the appropriate quantitation limit for the project. The remaining concentrations should correspond to the expected range of concentrations found in real samples or should define the working range of the GC. Each standard should contain each analyte for project completion (e.g., some or all of the compounds listed in Sec. 1.1 may be included). Volatile organic standards are prepared in organic-free reagent water.

10.8 Internal standards (if internal standard calibration is used) - To use this approach, the analyst must select one or more internal standards that are similar in analytical behavior to the compounds of interest. The analyst must further demonstrate that the measurement of the internal standard is not affected by

method or matrix interferences. Because of these limitations, no internal standard can be suggested that is applicable to all samples.

10.9 Surrogate standards - Whenever possible, the analyst should monitor both the performance of the analytical system and the effectiveness of the method in dealing with each sample matrix by spiking each sample, standard, and blank with one or two surrogate compounds which are not affected by method interferences.

11) INSTRUMENT CALIBRATION:

11.1 Initial Calibration Procedure

11.1.1 Inject 1 ul of each of the analyte levels utilizing the appropriate instrument operating conditions.
11.1.2 Record the peak area for each peak.
11.1.3 Establish the Calibration Factor (CF) for each standard level and evaluate the calibration curve as documented in Section 11.2. If acceptable, proceed to Section 11.2.5.
11.1.4 Analyze each of the remaining standards (Section 10.8).
11.1.5 Repeat Sections 11.2.2 and 11.2.3 for each standard.
11.1.6 Establish the CF for each analyte as documented in Section 11.3.
11.1.7 Establish the appropriate Retention Time (RT) window for each analyte as documented in Section 11.3.5.

11.2 Initial Calibration Curve

11.2.1 Calculate the calibration factor for each analyte at each standard concentration as documented in Section 15.

11.2.2 Calculate the mean calibration factor, and the relative standard deviation (RSD) of the calibration factors as documented in Section 15.

11.2.3 If the RSD for each analyte is < 20%, then the response of the instrument is considered linear and the mean calibration factor can be used to quantitate sample results. If the RSD is greater than 20%, then linearity through the origin cannot be assumed. The analyst must use a linear calibration curve or a non-linear calibration model (e.g., a polynomial equation) for quantitation.

11.2.4 For a linear calibration curve ($y = ax + b$), the analyst should not force the line through the origin, but leave the intercept calculated.

11.2.4.1 For an acceptable calibration curve, the coefficient ≥ 0.995.

11.2.4.2 If the approaches described above have not met the acceptance criteria, a non-linear calibration model may be employed. The quadratic (second order) model requires six standards.
$Y = ax^2+bx+c$

11.2.4.3 For an acceptable non-linear calibration, the coefficient of the determination (COD) must be $\geq$ 0.995.

11.2.5 Retention Time (RT) Windows

11.2.5.1 Record the retention time of each peak in the peak pattern.

11.2.5.2 Calculate the mean RT and standard deviation (SD) of each peak

11.2.5.3 The RT Window for each peak is defined at $\pm$ 3 times the SD around the mean RT.

11.2.5.4 If the SD of the retention time is 0, a default value of 0.03 minutes should be used to define the window.

11.3 Initial Calibration Verification

11.3.1 Verify each new Initial Calibration using a second source standard at or near the midpoint of the curve.

11.3.2 Agreement with the new curve must be $\pm$ 15 percent of the true value of the second source standard.

11.4 Continuing Calibration Verification

11.4.1 Utilizing a mid-level calibration standard, verify instrument calibration prior to any sample analyses, at intervals of not less than once every twenty samples, and at the end of the analytical sequence.

11.4.2 The calibration factor for each analyte must not exceed a $\pm$ 15 percent difference from the mean calibration factor calculated for the initial calibration. If a non-linear model has been employed for the initial calibration, % drift must be $\pm$15%. Refer to section 15 for calculation of % drift.

11.4.3 If the calibration does not meet the $\pm$15% limit, check the instrument operating conditions, perform any needed maintenance, and inject another aliquot of the calibration verification standard. If the

response for the analyte is still not within ±15%, a new initial calibration must be prepared.

11.4.4 Compare the retention time of each analyte in the calibration standard with the retention time window established in Section 11.3.5. Each analyte in each standard must fall within its respective retention time window. If not, the gas chromatographic system must either be adjusted so that a second analysis of the standard achieves acceptance criteria or a new initial calibration must be performed and new retention time windows established.

12) SAMPLE PREPARATION AND ANALYSIS

12.1 Sample Preparation

12.1.1 Water samples are prepared by separatory funnel extraction, method SW 3510, HS-EXT-001.

12.1.2 Soil samples are extracted by method SW 3540 (soxhlet) HS-EXT-002 or by 1:1 mix with water.

12.2 Sample Analysis

12.2.1 Qualitative identifications of target analytes are made by examination of the sample chromatograms and comparison of sample peak patterns versus the peak patterns established from individual standards.

12.2.2 Sample analysis must be bracketed with acceptable calibration verification standards. Should a calibration verification standard fail to meet QC criteria, all samples that were injected after the last acceptable standard must be re-analyzed.

12.2.3 Multi-level standards are highly recommended during the analytical sequence to ensure that detector response remains stable over the calibration range.

12.2.4 Sample injections may continue for as long as the calibration verification standards meet instrument QC requirements.

12.2.5 Use the calibration standards analyzed during the sequence to evaluate retention time stability. If any of the standards fall outside their established retention time windows, the problem must be corrected and all associated sample extracts re-analyzed.

12.2.6 If compound identification or quantitation cannot be completed due to matrix interferences (e.g., broad, rounded peaks or ill-defined baselines) cleanup of the extract or replacement of the capillary column is warranted.

12.2.7 Qualitative identification

12.2.7.1 Identification of analyte is based on the agreement of peak pattern, peak pattern retention time, and peak area ratios in the sample chromatogram with those established through the analysis of standards.

13) DATA ACQUISITION

13.1 Instrument operation and data collection utilizes HP ChemStation with Enviroquant data acquisition software.

13.2 Enviroquant data processing software converts the acquired signal information into final results.

14) CALCULATIONS & DATA REDUCTION

14.1 Calibration Factor (CF) and calibration RSD calculations:

14.1.1 Calibration Factor and Mean Calibration Factor:

14.1.1.1

$$CF = \frac{\text{Peak Area (or Height) of the Compound in the Standard}}{\text{Mass of the Compound Injected (in nanograms)}}$$

14.1.1.2 $$\text{Mean} \quad CF = \overline{CF} = \frac{\sum_{i=1}^{n} CF_i}{n}$$

where: n is the number of standards analyzed.

14.1.2 Standard Deviation (SD) and RSD:

$$SD = \sqrt{\frac{\sum_{i=1}^{n}(CF_i - \overline{CF})^2}{n-1}} \qquad RSD = \frac{SD}{\overline{CF}} \times 100$$

14.2 Calculation of Linear Regression Correlation Coefficient, r

$$r = \frac{\sum XY - \frac{\sum X \sum Y}{n}}{\sqrt{\left(\sum X^2 - \frac{(\sum X)^2}{n}\right)\left(\sum Y^2 - \frac{(\sum Y)^2}{n}\right)}}$$

Where:

X = individual values for independent variable
Y = individual values for dependent variable
n = number of pairs of data.
df = n-2

14.3 Calculation of % difference (using the calibration factors):

14.3.1 % Difference = [(CF - mean CF) x 100] / mean CF

where:
CF = the calibration factor from the CCV and
mean CF = the mean calibration factor from the initial calibration

14.4 Calculation of % drift (linear and non-linear regression) uses the following formula:

14.4.1 $\%\ \text{Drift} = \frac{[(\text{Calculated Conc.} - \text{Theoretical Conc.}) \times 100]}{\text{Theoretical Conc.}}$

14.5 Sample Quantitation using External calibration, aqueous samples:

$$\text{Concentration (ug/L)} = \frac{(A_x)(V_t)(D)}{(CF)(V_i)(V_s)}$$

where:

A_x= Area (or height of the peak) for the analyte in the sample.

V_t= Total volume of the concentrated extract (ml).

D = Dilution factor (if sample/extract was diluted prior to analysis)

CF= Mean calibration factor from the initial calibration (area/ng).

V_i= Volume of the extract injected (ml).

V_s= Volume of the aqueous sample extracted in L.

14.6 Sample Quantitation using External calibration, solid samples:

$$\text{Concentration (ug/kg)} = \frac{(A_x)(V_t)(D)}{(CF)(V_i)(W_s)}$$

where:

A_x= Area (or height of the peak) for the analyte in the sample.

V_t= Total volume of the concentrated extract (ml).

D = Dilution factor (if sample/extract was diluted prior to analysis)

CF= Mean calibration factor from the initial calibration (area/ng).

V_i= Volume of the extract injected (ml).

W_s= Weight of sample extracted in kg.

14.7 QC Calculations: Calculate the percent recovery for surrogates and for various QC samples (MS, MSD, LCS) according to the following equations:

14.7.1 Surrogate Recovery: Sample, matrix spike/matrix spike duplicate, duplicate, and blank samples are all spiked with surrogates prior to extraction. Surrogate percent recovery is calculated as follows:

$$\%R = \frac{SurrSR}{SurrSA} \times 100$$

where:
SurrSR = Surrogate Spiked Sample Result (µg/L or µg/kg).
SurrSA = Surrogate Spike Amount Added (µg/L or µg/kg).

14.7.2 % Recovery, %R (for MS and MSD Samples)

$$\%R = \frac{(SSR - SR)}{SA} \times 100$$

where:
SSR = Spiked Sample Result (µg/L or µg/kg).
SR = Sample Result (unspiked).
SA = Spike Amount Added (µg/L or µg/kg).

14.7.3 % Recovery, %R (for standards and LCS)

$$\%R = \frac{SSR}{SA} \times 100$$

where:
SSR = Spiked Sample Result (µg/L or µg/kg).
SA = Spike Amount Added (µg/L or µg/kg).

14.7.4 % RPD (for precision or replication evaluation)

$$\%RPD = \frac{|SR_1 - SR_2|}{½(SR_1 + SR_2)} \times 100$$

where:
SR_1 = Sample result for replicate 1.
SR_2 = Sample result for replicate 2.

15) Quality Control, Data Acceptance, & Corrective Action

15.1 Initial Calibration:

15.1.1 Frequency: A new curve must be generated when the ICV or CCV criteria are not met, after major instrument maintenance, or changes in operating conditions occur.

15.1.2 Acceptance Criteria:

15.1.2.1 Curve must contain 5 points minimally for the standard.
15.1.2.2 The mean RSD must be ≤ 15%, or
15.1.2.3 Establish calibration by least squares regression, or

15.1.2.3.1 COD must ≥ 0.995.

15.1.2.4 Establish calibration by quadratic (second order) model

15.1.2.4.1 Six standards minimally must be used, and
15.1.2.4.2 Coefficient of determination (COD) must be ≥ 0.995.

15.1.3 Curve Failure Corrective Action:

15.1.3.1 Verify standard integrity, perform any necessary instrument maintenance, and repeat calibration process.

15.1.3.2 All samples associated with a failed initial calibration curve must be reanalyzed prior to reporting. If reanalysis cannot be completed, all associated sample results must be flagged as "unusable" and narrated.

15.2 Initial Calibration Verification (ICV):

15.2.1 Perform evaluation each time a new curve is generated.

15.2.2 Verification must be completed against a second source standard. If an alternative supplier is not available, a different lot number must be used.

15.2.3 Agreement between the curve and the ICV results must be between 85 - 115 % of the ICV true values.

15.2.3 If the ICV fails to achieve acceptance criteria, evaluate standard integrity and/or perform any needed system maintenance. If a subsequent ICV analysis cannot achieve acceptance criteria, prepare new standards and/or generate new curve.

15.2.4 All samples associated with a failed ICV must be reanalyzed prior to reporting. If reanalysis cannot be completed, all associated sample results must be flagged as "unusable" and narrated.

15.3 Continuing Calibration Verification(CCV):

15.3.1 The CCV must be analyzed at the beginning of each daily sequence,minimally after every 20 samples, and at the end of the sequence.

15.3.2 All analytes must be $\pm$15 % of the expected value.

15.3.3. If the calibration does not meet the criteria, perform any necessary maintenance and re-analyze the standard. If the subsequent CCV fails to achieve acceptance criteria, prepare a new calibration curve.

15.3.4 All samples associated with a failed CCV must be reanalyzed prior to reporting. If reanalysis cannot be completed, all associated sample results must be flagged as "estimated" and narrated.

15.4 Retention Time (RT) Window:

15.4.1 Calculate new RT windows with each new curve.

15.4.2 The RT window must be set at $\pm$3 times the SD relative to the mean RT for each analyte.

15.4.3 Peak patterns for standards and QC samples must fall within the

documented RT window. (For field samples, analyst judgment may be substituted in situations involving chromatographic matrix interference.)

15.4.4 If RT acceptance criteria are not achieved, perform any necessary instrument maintenance and repeat the analysis. If the subsequent analysis fails to achieve acceptance criteria, perform a new initial calibration and establish updated RT windows.

15.5 Method Blank:

15.5.1 A method blank must be processed at a frequency of one per analytical batch of 20 or less samples. If the method blank indicates contamination, process an instrument blank to document instrument cleanliness.

15.5.2 Analytes of interest should be less than ½ the MQL and must be less than the MQL.

15.5.2.1 Other approved QA program requirements must be followed when the acceptable blank contamination specified in the approved quality assurance project plan differs from the above.

15.5.2.2 If blank contamination is present, the blank may be considered valid if the analytes of interest are less than 5% of the regulatory limit associated with an analyte or analytes of interest are less than 5% of the sample result for the same analyte, whichever is greater.

15.5.3 If the method blank results do not meet acceptance criteria, the contamination source(s) must be eliminated and all associated samples re-extracted. If samples cannot be re-extracted because of insufficient sample or other similar circumstances, a corrective action report must be initiated and issued to project management and to QA. The CAR must be detailed enough for preparation of the project narrative, and all appropriate data flags must be entered into the LIMS for the final report preparation. Data reported with an associated contaminated method blank must be flagged with a "B".

15.6 Laboratory Control Sample/Laboratory Control Duplicate (LCS/LCSD):

15.6.1 The LCS/LCSD must be processed with each batch of 20 or less samples utilizing a clean matrix.

15.6.2 LCS/LCSD accuracy must fall with 50% - 150%, and precision must fall within ± 25%.

15.6.3 If the LCS/LCSD recovery for the compounds of interest do not meet acceptance criteria, the sample batch must be re-extracted. If reprocessing it is not possible due to lack of sample or expired hold time, report (narrate) the variance to the client and flag the associated data as "estimated".

15.7 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

15.7.1 Samples should be spiked with the analyte mixture.
15.7.2 The MS/MSD should be processed with each batch of 20 samples or less (assuming sufficient sample volume).
15.7.3 MS/MSD recovery should fall within 50% - 150%, and the RPD must fall within $\pm$ 50%.
15.7.4 If the MS/MSD recoveries are outside acceptance criteria, the deviation may be related to matrix effects. In such instances, the LCS/LCSD and surrogate recoveries must carefully evaluated to determine if matrix interference is present or if method performance is poor. (Note that the MS/MSD are used to evaluate the matrix effect, not to control the analytical process.) If matrix interference is suspected, re-extraction is not necessary. If systemic error is suspected, all associated samples must be re-extracted.

15.8 Surrogates

15.8.1 Surrogates must be added to all samples. Recoveries must be evaluated for all samples and QC samples.

15.9 Demonstration of Proficiency:

15.9.1 Each analyst must demonstrate initial proficiency with sample preparation and/or determination by generating 4 sets of data of acceptable accuracy and precision for target analytes in a clean matrix.
15.9.2 Each analyst must demonstrate ongoing proficiency annually with sample preparation and/or determination by generating 4 sets of data of acceptable accuracy and precision for target analytes in a clean matrix, or by acceptable PE studies.

15.10 Method Detection Limits (MDLs) must be determined on each instrument on an annual basis (at minimum), or whenever major modifications are performed.

15.11 All deviations from documented acceptance criteria must be documented

using a Non-Conformance/Corrective Action Report (NCR/CAR) form and submitted to the QA Manager.

16) DATA AND RECORDS MANAGEMENT

16.1 All data shall be retained for a period of no less than 5 years and all electronic records for a period of no less than 7 years.
16.2 Hard copy documentation must be maintained for standard/chemical tracking, extraction procedures, maintenance, and run logs.
16.3 The primary analyst must review raw data after analysis and complete the review checklist. Any manual integrations or deletions must be dated and initialed by the analyst.
16.4 Instrument hardcopies must be maintained in daily batch folders that are instrument specific. The folder shall contain the associated sequence log, CCV summary sheet, all raw data w/quantitation report, and batch checklist.
16.5 Each batch must be peer reviewed by the department supervisor (or designee) prior to final reporting in the laboratory information management system (LIMS).

16.6 Pending data review and verification, all batch folders must be stored systemically in the instrument work area. After three months (or as defined by storage limitations), data must be transferred to the QA department for archival.
16.7 All data acquisition information must be stored on the computer hard drive and archived to CD format on a monthly basis.

17) Contingencies for Out-of-Control or Unacceptable Data

17.1 When QC failures occur, the source of the QC failure must be determined, corrected, and sample reanalysis carried out whenever possible.
17.2 When sample analysis cannot be repeated due to sample unavailability or holding time issues, data associated with failed QC data must be appropriately flagged and narrated.

18) Method Performance

18.1 Refer to table 20.1 for method performance data.

19) Reference Section

19.1 U.S. Environmental Protection Agency, "Method 8015D Nonhalogenated

Organics Using GC/FID", Test Methods for Evaluating Solid Waste Physical/Chemical Methods, Update 4 June, 2003.

19.2 U.S. Environmental Protection Agency, "Method 8000B Determinative Chromatographic Separations", Test Methods for Evaluating Solid Waste Physical/Chemical Methods, Update III, June 13, 1997.

19.4 ALS Laboratory Group Quality Assurance Manual, Revision 4 (or most current)

20) TABLES

20.1 Methd Calibration /QC Summary

Table 20.1 - Summary of Calibration and QC Procedures for Method SW8015			
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action**
Five-point initial calibration.	Initial calibration prior to sample analysis.	Average response - mean RSD for all analyte<20%; or Linear- r>0.990 or Quadratic - least squares regression $r^2 > 0.995$ (six points required)	Correct problem then repeat initial calibration.
Initial calibration verification	Once per five-point initial calibration. Required second source standard.	Mix within ± 15 % of expected value.	Correct problem then repeat initial calibration.
Retention time window	Each initial calibration and calibration verifications.	± 3 times standard deviation for each analyte relative to mean RT	Correct problem then reanalyze all samples analyzed since the last retention time check.
Continuing calibration verification	Daily, before sample analysis, after every 20 samples, and end of sequence.	All analytes within ±15% of expected value.	Correct problem then repeat initial calibration.
Demonstrate ability to generate acceptable accuracy and precision using four replicate LCS analyses.	Once per analyst.	QC acceptance criteria.	Recalculate results; locate and fix problem with system and then rerun demonstration for those analytes that did not meet criteria.
Method blank.	One per analytical batch.	No analytes detected >MQL.	Correct problem then re-prep and analyze method blank and all samples processed with the contaminated blank.
LCS/LCSD	One LCS/LCSD per analytical batch.	QC acceptance criteria.	Correct problem. Re-prep & analyze all associated samples in the affected analytical batch.
MS/MSD	One MS/MSD per every 20 project samples per matrix.	QC acceptance criteria,.	Describe in Laboratory Review Checklist.
MDL study.	Once per 12 month period.	Detection limits established shall be ≤ ½ the MQLs in table 21.1.	None.